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DEVELOPMENT AND EVALUATION OF INSECT REPELLANT PVC BASED FORMULATIONS

A MAJOR PROJECT SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE DEGREE OF

MASTER OF ENGINEERING
IN
POLYMER TECHNOLOGY

In the faculty of technology
University of Delhi, Delhi

By

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Under the Guidance of

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CERTIFICATE

This is to certify that Mr. JITENDRA SARIN from DELHI COLLEGE OF ENGINEERING, DELHI has individually completed a major project entitled "Development and Evaluation of Insect Repellant PVC Composition" under joint supervision of Dr. A.P. Gupta, Head, Department of Applied Chemistry and Polymer Technology, Dr. G.L. Verma, Asst. Professor, Depatrment of Applied Chemistry and Polymer Technology and Dr. M.Q. Parwez, Shriram Institute for Industrial Research, Delhi during session 2001-2002 in partial fulfillment of the requirement for the degree of Master of Engineering in Polymer Technology of Delhi University. This work has not been published anywhere for award of any degree.

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(JITENDRA SARIN)

SYNOPSIS

Development and evaluation of a PVC compound which is repellant to both termites and rats, was a valuable and extensive work conducted at Shriram Institute for Industrial Research, Delhi. During the project which was sponsored by M/s SPL Sidhartha, Noida (UP), a lot of literature survey as well as market survey was carried out and afterwards exhaustive experimental work (dry blending, roll milling, compression molding) and testing brought forward the valuable results substantially.

The effective PVC compound was required for making the PVC-Jute laminated floor coverings which may not be attacked by termites and rats as well. That's why all the testings were carried out on PVC-Jute laminates of specific sizes.

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1. INTRODUCTION:

1.1 A BRIEF OVERVIEW OF PVC

Poly (vinyl chloride) (PVC) is the product of bulk, suspension, emulsion or solution poylmerisation of vinyl chloride; out of which it is manufactured on large scale by suspension and bulk polymerization processes. It is a fine porous powder whose properties such as molecular weight, molecular weight distribution, chain structure, particle size and morphology etc., largely determine its behaviour in processing and service and the performance of products based on it. PVC is a solid, white colored material having a degree of polymerization between 100 and 2500 and a crystallinity of ca. of 10 weight percent. The elementary units of polymer chains mainly occupy positions 1,2. Morphologically, the grains of bulk and suspension PVC powders are classified into homogenous, nonhomogeneous and porous types the morphology emulsion polymerized PVC grains is substantially different from that of suspension polymers; the particles have been divided into cenospherical(hollow) particles and plenospherical(compact) particles the type of PVC grains required is determined by its intended use.

Vinyl chloride monomer (chloroethene, CH2=CHCl) is a gaseous, chlorine-containing hydrocarbon. The various PVC homopolymers and copolymers derived from it comprise a truly ubiquitous class of resins. In world wide commercial importance of PVC resins undoubtedly outrank every class or family of polymers both in diversity of application and in total tonnage of products made from PVC. Although PE may now outrank PVC in polymer synthesis and manufacture, PVC nevertheless hold a substantial edge over PE in terms of total finished products output,

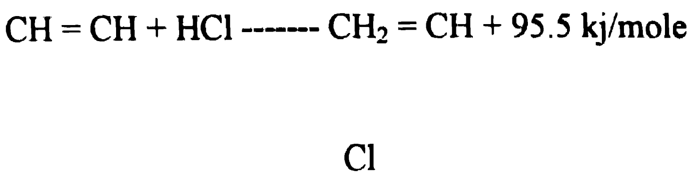
owing to the fact that PVC compositions generally contain large amount of modifying agents.

Although PVC was first recognized and characterized more than 100 years ago, it did not become a material of more than academic curiosity until 1920's and 1930's. It became commercially significant in Germany prior to World War II, and in USA it developed very rapidly during the war and in immediate post war period. Since then, it has had a marked growth curve.

It is an interesting paradox that one of the least stable of commercially available polymers should also be, in terms of tonnage consumption at least, one of the two most important plastics materials available today, Yet this is the unusual position hold by poly (vinyl chloride) PVC, a material whose commercial success has been to a large extent due to the discovery of suitable stabilizers and other additives which has enabled useful thermoplastic compounds to be produced.

After the war developments in Britain and the United States were concerned largely with plasticised PVC, handled mainly by extrusion, calendaring and paste techniques. Perhaps the greatest developments over the past few years have not been concerned with the molecular structure of the polymers but rather with the particles formed during polymerization. Such factors as particle shape, size, size distribution and porosity vitally affect the processing characteristics of the polymer and a more complete knowledge of their influence has led to many useful new grades of polymer.

For many years a major route to the production of vinyl chloride was the addition of hydrochloride acid to acetylene



$$CH = CH + HCl \text{------} \underset{Cl}{CH_2 = CH} + 95.5 \text{ kj/mole}$$

The acetylene is usually prepared by addition of water to calcium carbide which itself is prepared by heating together coke and lime. To remove impurities such as water, arsine and phosphine, the acetylene may be compressed to 15lbf/in2 (approx. 100 kPa), passed through a scrubbing tower tank chilled to – 10°C to remove some of the water present and then scrubbed with concentrated sulphuric acid. Hydrochloric acid may conveniently be prepared by combustion of hydrogen with chlorine.

As far as polymerisation is concerned, suspension polymerizations are generally easier to control and there is little, if any, loss in clarity or electrical insulation properties. Particle shape, size and size distribution may be controlled by varying the dispersing systems and the rate of stirring.

A typical charge would be :

Monomer	Vinyl chloride	30-50 parts
Dispersing agent	Gelatine	0·001 parts
Modifier	Trichlorethylene	0·1 parts
Initiator	caproyl peroxide	0·001 parts
	Demineralised water	90 parts

In addition, buffer salts such as disodium hydrogen phosphate may be used to prevent the pH of the aqueous phase falling during polymerization.

The main requirements of a flooring composition are that it should be hard, durable and competitive in price with other materials. This calls for highly filled materials, which are consequently harder to process than unfilled materials.

1.2 TYPICAL PROPERTIES OF PVC:

1.	Density at 293 K, g/cm^3	1.35-1.45
2.	Refractive index, n_D^{293}	1.544
3.	Flow point, K	450-495
4.	Glass transition point, K	351-378
5.	Heat conductivity, W/(m x K)	0.15-0.75
6.	Water absorption: In 24 hrs, %	0.4-0.6
	: In 1000 hrs, %	400(max.)
7.	Strength, MN/ m^2: Tensile	40-60
	: Compressive	78-160
	: Bending	80-120
8.	Elasticity modulus, GN/ m^2	3-4
9.	Impact strength (notched) KJ/m^2	2-10
10.	Yield strength, MN/m^2	10-30
11.	No. Average molecular weight (M_n)	50000-100000
12.	Degree of crystallinity	10-15%

13. PVC molecule is partially syndiotactic
14. PVC degrades beyond 200^0C with the evolution of HCL
15. Solubility of PVC

    Insoluble in methanol, ethanol, n-butanol, acetic acid, ether, water, formic acid, ethylene glycol, etc.

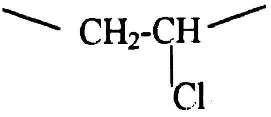
    Swelling in gasoline, CS_2, benzene, toluene.

    Soluble to some extent in chloroform, tri chloroethylene, acetone.

    Soluble in chlorohexane, nitrobenzene, pyridine, m-cresol, di chloroethane, THF(tetra hydro frane), methyl oxide.

Characteristics of PVC

The presence of the chlorine atom causes an increase in the inter chain attraction



And hence an increase in the hardness and stiffness of the polymer. PVC is also more polar than polyethylene because of the C-Cl dipole. Thus PVC has a higher dielectric constant and power factor than polyethylene, although at temperatures below the glass transition temperature (+ 80°C) the power factor is still comparatively low (0·01-0·05 at 60Hz) because of the immobility of the dipole.

The solubility parameter of PVC is about 19·4 $\text{Mpa}^{1/2}$ and the polymer is thus resistant to non-polar solvents, which have a lower solubility parameter. In fact it has very limited solubility; the only solvents that are effective being those, which appear to be capable of some form of interaction with the polymer. It is suggested that PVC is capable of acting as a weak proton donor and thus effective solvents are weak proton acceptors. These include cyclohexanone and tetrahydrofuran. There are many materials that are suitable plasticisers for PVC. They have similar solubility parameters to PVC and are also weak proton acceptors. These are of too high a molecular weight and too large a molecular size to dissolve the polymer at room temperature but they may be incorporated by mixing at elevated temperatures to give mixtures stable at room temperature. The presence of chlorine in large quantities in the polymer renders it flame retarding. The presence of plasticisers however reduces the resistance to burning.

X-ray studies indicate that the vinyl chloride polymer as normally prepared in commercial processes is substantially amorphous although some small amount of crystallinity (about 5% as measured by X-ray diffraction methods) is present. It has been reported that examination of the crystalline zoned indicated a repeat distance of 5·1Å which is consistent with a syndiotactic (i.e. alternating) structure. Later studies using NMR techniques indicate that conventional PVC is about 55% syndiotactic and the rest largely atactic in structure.

Commercial PVC polymers are largely amorphous, slightly branched molecules with the monomer residues arranged in a head-to-tail sequence. Individual grades of material do however differ in average molecular weight, molecular weight distribution, particle shape, size and size distribution, and in the presence of impurities. Some grades may also contain small quantities of comonomer residues.

For commercial purposes the molecular weight is usually characterized from measurements of the viscosity of dilute solutions.

1.3 APPLICATIONS OF PVC:

PVC compounds may reasonably be considered as the most versatile of plastics materials; their uses range from building applications to toys and baby-pants.

Plasticised PVC has been used for electrical insulation for many years. Although of only limited value in high frequency work it is of great value as an insulator, for direct current and low frequency alternating current carriers. It has almost completely replaced rubber insulated wire for domestic flex and is widely used industrially. PVC sheathing is widely used in cables where polyethylene is employed as the insulator.

PVC compounds, particularly unplasticised grades are extensively used in chemical plant. It is necessary, when considering PVC compounds for applications in which contact with chemicals will occur, to check that all of the ingredients will be resistant to them and also that they will not be leached out by them. Ingredients of the PVC compound must not affect the nature and properties of the chemicals. Unplasticised PVC is particularly use ful in acid recovery plant and in plant for handling hydrocarbond, many of which adversely affect the polyolefins.

Unplasticised PVC, which when carefully compounded and processed has excellent resistance to weathering has a great potential important building industry, it is now becoming used increasingly in place of the materials for many uses since, when the sost of installation is that account, it is frequently cheaper. Important used include guttering piping, conduit, window frames that neither corrode nor rot, and tarrace roof sheeting with good flame retarding properties. Plasticised PVC backed by such a variety of materials as plywood. Chipboard as concrete and aluminium is of value for wall cladding for both interior exterior application. One important

development has been the increment of UPVC as a wood replacement where the economies, taking into both initial cost and installation, are becoming more favourable over synthetic material. Specific applications include shiplap wall clock fencing, bench-type seating at sports stadia and window frames.

As a pipe material PVC is widely used in soil pipes and for drainages as for the above-ground applications Piping diameters of up to 60 cm is no longer uncommon.

In car applications PVC leathercloth has been widely used for many years in upholstery and trim.

Other transportation uses include the flexible rear window of sports cars, car covers and tool bags. PVC adhesives, generally contains polymerisable plasticiser, are finding useful outlets in car manufacture.

In house and other furnishings PVC compounds find many outlets PVC leathercloth is now generally accepted in kitchen upholstery.

Developments in paste and powder techniques have led to important made finishing applications for example in stacking chairs. Edging strip is widely used in office furniture. Washable wallpapers are obtained by treating that paper with PVC compounds.

The importance of PVC in packaging has increased continually. All PVC sacks, like the all-polyethylene sacks, enable fertilizers and other products to be stored out of doors. Unplasticised PVC bottles have better clarity and resistance and barrier properties than those made from polyethylene as compared with glass are of course less brittle and lighter and have great design flexibility. As a result PVC bottles have been widely used for consumable liquids (such as edible oils, fruit squashes, table wine and vinegar),

There are a number of examples of PVC used in personal apparel. Ladies' handbags are frequently produced from PVC leathercloth whilst `plastic rainwear' and baby-pants are made by high frequency welding of calendered plasticised PVC sheet. All-PVC shoes are of value in beach wear and increasingly for standard footwear. PVC is now proving to be an excellent abrasion resistant shoe-soling material. The sole may be applied by injection moulding. Paste injection moulding and adhesive techniques. PVC is also used in beachwear, in playballs and in babies' soft dolls. Other applications can be enlisted as:

1. Bulding and construction
    a. Lightening
    b. Pipes and fittings
    c. Profiles and conduits
    d. Rain water system
    e. Siding
    f. Swimming pool liner
    g. Water stops
    h. Weather stripping
    i. Windows
2. Clothing
    a. Baby pants
    b. Outer wears
3. Coatings
4. Electrical (wires and cables)
    a. Automotive
    b. Communications
    c. Constructions

5. Floorings
    a. Flooring sheets
    b. Flooring tiles
    c. Embossed carpet
    d. Earth flooring
6. Home furnishings
7. Packaging
    a. Blow molded containers
    b. Blistered packed pills
    c. Food
8. Phonographic records
9. Toys
10. Transportations
11. Miscellaneous
    a. Agriculture
    b. Credit cards
    c. Laminates
    d. Medical tubings
    e. Sporting goods
    f. Stationery supplies
    g. Tools and hardware

1.4 TOXICOLOGY AND OCCUPATIONAL HEALTH

VCM (vinyl chloride monomer) at ambient temperature is gas, boils at -13°C, so it is handled as a compressed liquid. It has a vapour density greater then air so pre cautions are taken in design not to contain the vapour in restricted areas.

Protection of reactors by relief valves or bursting discs and double valve isolation of all lines containing VCM are basic precautions taken in the design and construction of any PVC plant vessel, which contains monomers. The gas is explosive between 3.6 to 25% volume with air and 12-volume percentage oxygen is required for ignition. Therefore, a special series of working conditions must be satisfied in working environment and great care is always taken to purge lines, pumps, valves etc., before any maintenance work is started.

VCM has a narcotic effect at 8 to 12 % concentration and can cause death at higher concentrations. Exposure to VCM has been shown to lead to two distinct problems. In mid 1960's it became clear that a number of workers were effected by acroosteolysis (a disease related to bones of hands and feet's) due to long exposure to VCM of high concentration. Similarly in 1971 a new disease angiosarcoma (a rare kind of liver cancer) was observed. This knowledge has led to the introduction of vary stringent controls by governments and regularity authorities to limit the exposure of VCM to workers.

Strict limits have been set for the quantity of residual VCM in processed PVC articles intended for use in food contact applications.

Various plasticizers and solvents emissions and the dusting propensity of many finely divided powder ingredients, particularly those based on

substances considered to be of themselves highly toxics, e.g., powdered lead and cadmium containing stabilizers, pigments that also content these metals and also pigments containing Hg, Se, Cu, Cr, Ba, Ti. Vary finely divided carbon black is another frequently mentioned offender, although more for aesthetic than health reasons.

As PVC based material are highly resistant to bio degradation and to ultimate breakdown following their being discarded or buried either in land or in sea. When buried, it will give off highly poisonous and corrosive fumes.

There is little evidence that PVC powder itself causes any significant medical problems but steps are always taken to reduce powder emissions because of its nuisance value.

1.5 DISPOSALS AND RECYCLING:

Options for disposal of PVC include recycling, landfill and incineration. Today, most of our PVC waste comes from packaging. Although PVC has seen its greatest use in the construction industry, building products are long life, so the majority of PVC is current use. There is not yet a serious problem in PVC disposal. However, PVC waste may become a concern when buildings are refurbished or demolished. In Europe and USA, PVC makes a significant contribution to chlorine, cadmium and lead in municipal landfills.

Incineration of PVC is not widely acceptable as there is some doubt over its safety due to the potential emission of hydrochloride, heavy metals and dioxins, particularly in poorly designed or maintained incinerators. In the case of accidental fire, PVC has the advantage of being inherently non-inflammable. Rigid PVC will burn only with the continuous addition of heat from another source. Flexible PVC may continue burning after the external heat source has been removed, due to the addition of plasticizers, which lower the overall chlorine content of the material.

Recycling is the preferred option. PVC can be readily identified in the waste stream and separated for recycling. Once waste PVC is clean and separated from other plastics it is mechanically ground and mixed in with virgin PVC at a ratio of 10 to 15% without affecting quality. If cleaning is needed, the reprocessing consumes about 10% of the energy required to make virgin PVC. Currently, post-consumer and industrial PVC waste is being recycled into product such as hose core, car mud flaps, doormats and pipefitting. The industry is supporting efforts to expand recycling for non-packaging, such as construction and demolition scrap.

1.6 THE FUTURE OF PVC:

The PVC manufacturing industry has altered its processes and products over the years to meet an ever more stringent range of health and environmental safety standards, imposed by environment protection agencies worldwide. The Australians plastics and chemical industry association is moving to self-regulation with high-level environmental management systems.

Nevertheless, PVC is a highly versatile, durable and affordable plastic. The CSIRO found that although little detailed comparative information is available the balance of evidence suggests that there is no alternative material to PVC in major building product applications, which has less effect on the environment.

2. ADDITIVES FOR PVC:

The large number of additives used in polymer technology complicates the problem of accurate identification and analysis. A given plastic specimen may contain a verity of different additive such as plasticizers, stabilizers, anti oxidants, fillers and colorants that must be studied carefully for the polymer used and for the effect on one another.

2.1 STABILIZERS:

Stabilizers are added to PVC composition to protect against degradation to which the polymer is prone to, as under the influence of heat and mechanical shear in processing and also to afford longer-term protection in service, mainly against the effects of heat, light and oxidation.

The resin PVC when decomposing will discharge HCl and produce poliene. And this poliene is responsible for the color changing, and the decomposion will progress with chain reactions. For this reason, the stabilizers on found in the market include lead, cadmium, barium and strearic acid which will stop the progress of decomposition by chain reaction. Octane acid metal soap, organotin compounds which will eliminate poliene, epoxy compounds and various other organic and inorganic light stabilizers. Stabilizers are selected according to the intended uses of the product.

Despite the uncertainty regarding the actual detailed mechanism, the thermal degradation of PVC is known to include-

1. Dehydrochlorination of the chains through the elimination of HCl.

2. Formation of double bonds and conjugated double bond systems largely as a result of dehydrochlorination: the conjugated double bonds are primarily responsible for the development of the color.
3. Chemical changes at the principle points of vulnerability in the PVC chain i.e., the tertiary chlorine atom and the end groups.

REQUIREMENTS OF THE STABILIZERS:

A stabilizer supposed to have following desirable properties as

- Absorption of HCl.
- Displacement of labile chlorine atoms.
- Disruption of double bonds.
- Anti oxidant protection.
- Free radical absorption.
- Reactivity with resin impurities.
- Prevention of degradation products and
- Ultraviolet screening

In addition, from the standpoint of processing and use, the ideal stabilizer should also

- Be readily dispersible in the PVC compound at fully compatible with all constituents even after prolonged service.
- Have no adverse effect on processing properties.
- Be equally effective in PVC resins of all types and from all sources and
- Be inexpensive and effective in small proportions.

ACTIONS AND CHARACTERISTICS OF LEAD STABILIZERS:

Partly due to there low cost and partly to general effectivity and simplicity in formulation, these old established stabilizers are still widely used. They are particularly popular in PVC compound for electrical insulation, apart from it they also confer excellent resistance to both heat and light. Lead stabilizers function because of their acid accepting ability and the chloride of lead are insoluble and non-ionizable and therefore probably inert. Depending on structure of specific lead salts, some function as efficient white pigments, whereas with others, the best degree of transparency that can be obtained is a cloudy and milky transparency. Lead stabilizers are generally considered unsatisfactory because maintenance of color uniformity and freedom from slight color drifting during long continuous runs cannot be attained, changes in opacity developed with in the stock as stabilizers consume also change the tinting strength or pigment balance.

However, for applications in wires and cables and phonograph records these limiting characteristics are not considered serious drawback. The examples of specific lead stabilizers are described below:

1. Basic lead carbonate $2PbCO_3.Pb(OH)_2$ (known as white lead)
2. Litharge PbO
3. Basic lead silicate $3PbO.2SiO_2.2H_2O$
4. TBLS (Tri Basic Lead Sulphate) $3PbO.PbSO_4.H_2O$: Provides good heat stability and electrical properties without any danger of gassing. It is used in building wire compounds (60°C) but it is replaced with more economical basic lead silicate / sulphate.it is however too reactive for use with polyester plastizers to meet the aging requirement of high temperature (90°C-105°C) insulation.

5. Basic lead silicate sulphate
6. Dibasic lead phthalate
7. Dibasic lead phosphate
8. Dibasic lead strearate.

DRAWBACKS OF LEAD STABILISERS:

All lead stabilize vinyl composition are prone to sulphide staining when exposed to industrial atmosphere rich in hydrogen sulphide fumes. They will gradually take a chocolate brown to black appearance. Some times toxicological characteristics of lead salts may also be harmful.

Additional cleaning in plant resulting from their propensity towards dusting, their relatively high specific gravity and efficient lead stabilizers usually requires high concentration of additive then is the case with other stabilizing materials, most often an order of 5-8 phr.

ADVANTAGES OF LEAD STABILISERS:

Low cost performed higher degree of light stabilization; lead stabilizers are insoluble and therefore nonextractable from PVC composition. In vinyl foam formulations expanded by decomposition of azodicarbonamide, lead stabilization is advantageous because basic lead salts also are effective promoters and catalysts for the decomposition reactions of blowing agent thus making expansion possible at lower temperatures.

POSITIVE CONTRIBUTIONS OF VINYL STABILIZERS:

1. Heat stability: main contribution of vinyl stabilizers is improving heat stability. Improved heat stability is a very broad concept, to some, the improvement of the compound's initial color, after processing, is the most important criterion by which stabilizers performance is evaluated. To others minimal differences in original color are less important than the capacity of the stabilizers for holding that original color for extended periods of time, at elevated temperatures without allowing any color deviation or shift. "Creep" resistance in unplasticised siding and pipe is a property that partly depends on stabilizers selection and performance. In vinyl floor tiles, dimensional stability in both longitudinal and transverse direction is of critical importance. Development and maintenance of optimum mechanical properties, tensile strength degree of elongation, hardness and low temperature embrittlement are just some of the properties that can be affected. Stabilizers have marked effect on chemical and electrical resistance characteristics of vinyl materials.
2. Rheology: Effect of stabilizing agents on rheology of formulated PVC, in particular rigid PVC, vary quite widely, depending on amount of stabilizers additive added and specific chemistry of stabilizers systems. For individual metallic and organo metallic salts or complexes, there are separate and distinct contributions that arise from choice of cation as opposed to selection of anionic ligand. Each of so called organic "synergistic" and various diluents, coupling solvents and so fourth that used in stabilizers

manufacture makes additional contribution to the final rheological state of total PVC formulation. Magnitude of rheological contributions made by each separate ingredients can be quite large, though these substances are added in negligible quantity to total formulation. Rheological effect of stabilizers on vinyl compounds during mixing and processing are not always positive. Though stabilizing agents may be efficient from maintenance of desired properties point of view it may be worth less if negative rheological effect to great that processing of compound is severely retarded or rendered impossible.

3. MECHANICAL PROPERTIES: Ideal stabilizers should remain inert in the system, i.e., it should not influence the physical characteristics of the composition. Lead based stabilizers (insoluble solids), behave much like fillers being used in lower concentration. Except for majority of lead salt, most stabilizers are either wholly or partly soluble and their effect on various compound properties must frequently be taken into account and compensated for. Stabilizers can exert a plastisizing function and hence can influence properties as elongation, hardness, tensile strength and tear strength. In rigid compounds many of the most efficient stabilizers are very highly plasticizing with consequence adverse effect on heat distortion temperatures and brittleness(impact strength). True polymeric stabilizers are not always as high in compatibility as there lower molecular weight oligomers. When stabilizer is too immobile, its stabilizing may be impaired. Polyfunctional stabilizers (either as simple, low molecular weight materials or as true polymers), because of there

poly functionality are capable of reacting with and cross bridging adjacent PVC molecules and then become undesirable modifiers of the polymer system rheology.

4. ELECTRICAL PROPERTIES: For formulating, compound to be used as electrical insulation, it is necessary that compound be free of ionic material and particular attention be given to choice of stabilizers. Here principle functions of stabilizers are not to guard against color development but to ensure that end results of any degradation that may occur, either during processing or thereafter, will be presenting the form of nonionic degradation products. If stabilizer cannot provide this function, it would ineffective in protecting against loss of electrical properties, as build up of electrically conducting chlorides and other ions would contribute to significant loss of volume resistivity and other insulation qualities.

Lead salts are the most preferred electrical grade stabilizers as only chlorides of lead are water insoluble and nonionic compound. As a result some form of reactive lead compound rests as the most suitable, practical source of HCl sinking electrical insulation. Other metallic stabilizers are less suitable since thin cations are all conductive and chloride formed by these metals are both water soluble and ionic conductors in less demanding applications. Use of lead barium systems or lead calcium systems has been proposed in low voltage application where considerable color-coding of insulation is involved, or in transparent insulation, barium cadmium stabilizers are used. Electrical characteristics of powdered metallic stabilizers are improved by applying different

type of surface coating to stabilizers particles where rigid control of color is of secondary concern; we can obtain good electrical characteristics with all organic stabilizers.

Potential problems associated with stabilizers are:

1. Staining
2. Incompatibility
3. Exudation
4. Plating accumulation ("plate out")

2.2 PLASTICIZERS:

A plasticizer is a substance, which is added to material(usually a plastic, resin or elastomer) to improve its processibility, flexibility and stretchability plasticizer can decrease melt viscosity glass transition temperature and the modulus of elasticity of the product without altering the fundamental chemical character of the plasticized material.

The great advantage of the external plasticizing lies in its breath for variation, allowing regulation of the degree of plasticizing and making in practice, the most important method of plasticizing. A disadvantage of external plasticizing is the extractability of plasticizers from the plasticized material. This depends on contact medium but can never be completely avoided.

The principle of internal plasticizing involves monomers leading to homo polymers with high glass transition temperature being selectively co polymerized with monomers whose homo polymers have a substantially

lower glass transition temperature. The advantage of internally plasticized products lies in the strong chemical combination of hard with soft segments, which cannot be separated from each other by extraction. However the techniques are limited; every co polymer is only suited to certain flexibility requirement. The mechanical properties of internally plasticized materials show marked temperature dependence and the material have insufficient dimensional stability at elevated temperature. Of course, internally plasticized polymers can be plasticized externally as well.

Plasticizers may be defined in two ways. Firstly by reference of their chemical nature, and secondly by the function they perform. Generally, plasticizers are high boiling chemicals that may be liquids or solids, usually the former. As far as a functional definition is concerned, plasticizers convert the hard, horny PVC resins into flexible and workable materials. They also modify or extend the basic properties of the resins, or endow the resulting compounds with new properties.

Plasticizers may be divided into two main groups; primary plasticizers and secondary plasticizers. Primary plasticizers must get polymers in the usual processing temperature and highly compatible with the resins. They can be readily used alone. The secondary plasticizers are less compatible with PVC resins and are usually employed together with the primary plasticizers to confer some special property. The extenders from a branch of this group; most have limited compatibility and are frequently included in plasticized composition, as part replacement for other plasticizers, to reduce cost. Most of the compounds in common use as extenders are chlorinated paraffins or hydrocarbon oils.

THEORY OF PLASTICISATION:

As in other polymers, the molecular chains in PVC are held together by attractive forces arising as a result of the chemical structure and morphology of the polymer. These forces are reduced, and the molecular chain enabled to slip past one another more easily, when plasticizer molecule are incorporated between them. However, forces of the same nature operate also between the plasticizer molecules themselves, as well as between those of plasticizer and polymer. The plasticized polymer is thus a system in complex dynamic equilibrium, its properties being determined, in the main by plasticizer concentration and by the defects of plasticizer-polymer, polymer-polymer, and plasticizer-plasticizer interaction on the molecular scale. As is now realized, plasticization can only occur when the orders of magnitude of these interactions are all approximately the same the above remarks apply to externally plasticized PVC.

EFFICIENCY OF PLASTICIZERS:

It is obviously of the greatest importance to plastic industry to know which are good plasticizers. There is no standardized definition, but the term is usually held to be concerned with the following general requirements:

1. Compatibility and ease of mixing with the polymer.
2. Effectiveness in imparting a desirable property or properties to the plasticized polymer.
3. Permanence.

A good plasticizer is, therefore, one which fulfills simultaneously all the three principal requirements to the degree necessary in the particular application. In practice, the choice is almost invariably a compromise govern

mainly by the processing technique to be used, the end application of the plasticized material and economic considerations.

TYPES OF PLASTICIZERS:

1. Phthalate plasticizers (Diisooctyl phthalate)DOP
2. Phosphate plasticizers (trioctyl phosphate)TOP
3. Low-temperature plasticizers
4. Polymeric plasticizers (polypropylene adapate PPA & poly propylene sebacate PPS)

PLASTICIZER'S EFFECT ON MELT VISCOSITY:

The melt viscosity of PVC compounds decreases continuously with improved flow ability as the plasticizer content is raised. Even low plasticizers concentrations, which still lie in the brittle region, are clearly effective. Optimum flow properties improve manufacturing efficiency and processing technique such as, deep drawing or embossing.

PLASTICIZER'S EFFECT ON HARDNESS AND OTHER MECHANICAL PROPERTIES:

The hardness of polymers is reduced by plasticizing. The degree of effectiveness of plasticizers is polymer specific and also dependent on plasticizer structure, quantity and temperature. With PVC, after the brittle region has been passed, a soft, flexible product is obtained; increasing quantities of plasticizer cause decrease in hardness and depress the glass transition temperature from 80°C to about -50°C. Also with increasing plasticizer content the tensile strength decreases and the elongation increases.

2.3 FILLERS:

The modifying agents or additives used to adjust cost and reinforcement mainly are known as "fillers". In some cases the fillers added can improve some properties like reinforcement, hardening, improved slip or skid resistance, and better dry space and in drapery and upholstery fabrics. Such fillers are known as Functional Fillers. In actuality, fillersmust be choosen with great care because of affect they can have the total system. In some cases the filler can be up to 80 weight % of the total system. Fillers such as carbon black dramatically increase the tensile strength of a system. Clays and talc, with their low cost can reduce the overall cost of a system. However, cost reduction at the expense of final properties leads to a system that cannot pass the required tests and is therefore worthless. Various chemical properties of fillers , such as alkalinity of clays, can have an effect on the overall system stability or performance.

REQUIREMENTS OF FILLER:

1. The fillers should have high standard of uniformity. As much care should be exercised on it as on the plasticizers and stabilizers.
2. It should be as wide as possible, free from foreign matter, be nonabrasive and should not contain chemical impurities.
3. Particle should be small (not more than 3 to 5μ) and uniform.
4. The processing property should be good. In particular, it should be easy to disperses and not cause sticking to mill or calendar.
5. The plasticizer absorption should be low.
6. It should not adversely affect the stability of the compound.

CARBON BLACK:

There are literally hundreds of grades of carbon black, each designed to give specific properties the differences are generally; particle size, particle geometry, or structure, and oil content. The characteristic for the various grades are defined in AASTM D1765 and ASTM D3053. Carbon blacks are primarily used for reinforcement. However, they also give UV resistance and abrasion resistance.

MINERAL FILLERS:

$CaCO_3$

It has an extremely favorable balance of properties that recommended as filler and is among the least expensive mineral fillers. It is a clean, neutral white and thus lands itself well to pigmentation in any direction.

Advantages of $CaCO_3$ as filler:

1. It is freely available, thus it does have to be transported over great distances.
2. The stone is amenable to grinding to low particle size, thus it is generally available in wide range of particle sizes and particle-size distribution specifications.
3. It is relatively soft mineral and if not contaminated with silica, has a relatively low abrasion rating.
4. It has a relatively low specific gravity (~2.71), and because of this has an advantage in pound-volume cost.

   Calcium carbonate is perhaps the lowest cost compounding ingredient. It also gives a smooth surface to a compound, for these reasons it is used extensively as filler. Calcium carbonate does not

have some disadvantages however, because of its spherical particle geometry it does not significantly reinforce the compound, and because of its high alkalinity it can interfere with halogenated flame retardants. Calcium carbonate should not be used in systems containing halogenated flame retardants.

With fluidized bed formulations, finely powdered additive such as $CaCO_3$ absorbs the excess plasticizer on the vinyl resin and behave as miniature ball bearings between the larger resin particles. These filler additive dry the mixture prevent agglomeration, and help to de agglomerate existing clusters.

Other examples of minerals are

-asbestos

-clay, talc and related silicate ores

-silica

-Miscellaneous- alumina, barytes ($BaSO_4$), and gypsum ($CaSO_4$)

NON MINERAL FILLERS:

They widely consist of liquids or gases or both and a few solid particles. They consists of a number of fibers and flours, wool, glass, cellulose, wood flour(sawdust), ground walnut and pecan shells, jute, sisal, nylon and rayon.

FUNCTIONAL FILLERS:

These fillers in addition to acting as the cheap agent also improve some properties like reinforcement, hardening, improved slip and skid resistance etc. examples of such fillers are - ppt. metallic silicates, ppt. silica gel, pyrogenic silica, ppt. $CaCO_3$ (cause less surface mar and crease whitening than ground one and leads to an a increase in impact strength at

low concentration of ~5 to 20 phr and also sometimes acts as secondary heat stabilizer), antimony oxide, carbon black, iron oxide, zinc oxide.

FACTORS AFFECTING PERFORMANCE OF FILLERS IN VINYL COMPOUNDS:

a. COLOR AND TINT STRENGTH:

The filler must be as transparent as possible and at same time be uncolored for it to be not visually discernible.

b. EFFECT OF REFRACTIVE INDEX:

As the refractive index of the fillers gets further and further from that of the weighted average of all the refractive indices of the individual components (resin, plasticizers, stabilizers, etc.). The individual stress whitening is due to a discontinuity in refractive index with in the heterogeneous system, since under the mechanical force of stressing the material, a partially separation of phase occurs, producing minute voids or empty spaces. These have a different refractive indices and the light waves passing through these regions are bent accordingly, thus producing the typical whitening appearance this type of stress whitening occurs only when the filler has not been sufficiently wetted by the polymer plasticizer network and resulting bonding is not too strong.

c. EFFECT OF PARTICLE SHAPE, SIZE AND SIZE DISTRIBUTION:

These three factors will influence the degree of gloss air entrapment, permeability, and fire resistance, as well as mechanical properties (tensile strength, elongation and

hardness), and dimensional properties (creep and shrinkage) of vinyl formulations. The same three factors also significantly affect the rheological properties and hence the processing characteristics of vinyl compounds. Problems such as low out put, machine hang up(with resultant burning and degradation of the stagnating compounds), pinholes and blistering can all be attributed on occasion to a faulty selection of filler particle size, size distribution, or shape.

d. SURFACE AREA AND OIL ABSORPTION:

Porosity and shape of the particle largely control the rheology of binder-filler system. As the porosity increases so does the surface area, which in turn helps to increase the oil absorption or binder requirement of the fillers. Asbestos and coarse dry ground limestone illustrate the extremes. Fibrous asbestos with high porosity requires about twice the binder levels of relatively nonporous cores limestone to obtain approximately equivalent melt rheology of the vinyl filler composite.

e. FILLER MODULUS:

It should be noted that with good adhesion of polymer to filler, much greater reinforcing action of the continuous phase can be expected when the filler modulus greatly exceeds the matrix modulus, compared with when they are equal or the filler modulus is only slightly greater. When there is poor adhesion, the modulus can decrease.

f. SURFACE TREATMENT:

Surface treatment of fillers can alter the degree of polymer-filler adhesion and thereby influence by the filler e.g., silicon-coupling agents have been used with silica fillers, such as glass fibers, wollastonite clay, and asbestos. It is also observed as some benefits of silicon coupling agents when they are used with CaCO3 filler.

g. ABRASION AND HARDNESS:

The harder fillers are found to be more abrasive on processing rolls, screws, cylinder walls, dies, moulds, mixer blades etc. so they have an unfavorable economic aspects arising from the need periodically to replace or rebuild critical items of processing equipments.

Also the continual abrasion of the metallic surfaces results in compound contamination. This usually results in two fold effect-(1) discoloration due to the inadvertent pigmentation effect of the metallic contaminant, and (2) premature and unanticipated heat- stability failure, resulting from the catalytic degradation influence of heavy- metal contaminants(usually iron, steel or copper).

2.4 LUBRICANTS:

A lubricant is added to PVC compounds:

(I) Reduce the friction (and adhesion) of compounds against working surfaces in processing.

(II) Lower the inter- particle and inter-molecular friction in processing which reduces the effective melt viscosity and heat build-up.

CLASSCIFICATION OF LUBRICANTS:

EXTERNAL LUBRICANTS: - These lubricants promote slip and release from the metal surfaces of processing equipment. They reduce the affinity of the plastic melt for the metal surfaces. They have a higher affinity for the metal and very low compatibility in the plastic melt, establishing a barrier between plastic and metal e.g., paraffin oil, hydrocarbons, n-paraffin, polyethylene wax.

INTERNAL LUBRICANTS: - They reduce melt viscosity and consequent functional heat build up and are considered as pseudoplasticizers. They are generally more compatible with PVC compounds than external lubricants. Structurally, internal lubricants are composed of one or more long chain hydrocarbon segments together with a polar functional group. The later is required for proper balance of compatibility and efficiency at melt temperatures and to prevent exudation at ambient temperatures e.g., mono fatty acids or glycerol, fatty alcohols, and fatty acid ester waxes such as castor and stearic acid etc.

Compatibility of a lubricant is governed by a balance of polarity of the functional groups present and the hydrocarbon chain length. Accordingly, the chemical structure is the primary factor that determines weather the lubricant will act externally or internally. It appears that ideal lubricants should be built up of as long a hydrocarbon chain as possible linked to a terminal group giving a large dipole moment. Stearic acid is more lubricating than Lauric acid because of its longer chain length and lower polarity and compatibility.

Metallic soaps have been generally chosed as internal lubricants. This is true for calcium stearate; but lead stearate and particularly dibasic lead stearate are much more external in mode of lubrication. Paraffin and polyethylene waxes are classified as external lubricant because of their low compatibility. However, in the presence of some impact modifiers like ABS, MBS and acrylics, with which they have much grater compatibility, they start acting as internal lubricants.

PROPERTIES AFFECTED BY LUBRICANTS:

A. During Processing: -

1. The lubricant has marked effect on fusion, particularly in unplasticized PVC. Fully dispersed external lubricants will coat the resin particles and retard their coalescence and fusion physically by minimizing the development of frictional heat. The external lubricant coating will also retard the inter diffusion of the short chain segments of the polymer chain.

2. The lubricant ill also affect the temperature of the PVC melt. External lubricants, since they retard and reduce shear heat, will given slower rate of temperature rise then internal lubricant. The lubricated composition has its melt temperature reduced by either type of lubricant in contrast with an unlubricated composition.
3. The rheology of the PVC is obliviously effected by the lubricant. Flow characteristics are governed by the rate of fusion, degree of fusion and melt viscosity and by adhesion or slip on metal surfaces of the processing equipment. Melt fracture or flow instability, generally considered to be caused by the elastic nature of the polymer melt and their resultant turbulent flow, can also be affected by lubricant balance.
4. Use of excessive quantities of external lubricant, produces undesirable effects such as slippage and surging. Also because of the limited compatibility of external lubricants, use of excessive levels will cause plating problems. Not only will the lubricant plate out on the metal surfaces but also it will carry with another components leading to streaking and burning of the compound. Also the excess quantity of internal lubricant e.g., stearic acid causes the compounded PVC insulation to have improper adhesion with the conductor.

Considering the influence of lubricant on the preceding factors, it is obvious that lubricants can affect processing rates.

B. Finished products:

1. Lubricant can effect the mechanical properties such as tensile strength, elongation, and impact strength e.g., partially oxidized poly ethylene in certain formulation of rigid PVC offer improved ductility. Some ester waxes, such as glycerol tristearate are also useful.

   When normal lead stearate is used as the sole lubricant, higher impact strength is obtained with an increased level. This is unexpected and may be due to laminated composition at the higher levels. Dibasic lead stearate shows an opposite trend. Normal lead stearate is a very active lubricant and small quantities are sufficient to cause processing difficulties. Dibasic lead stearate is less active and doesn't cause this difficulty. A blend of the two lubricants provides flat impact strength.
2. Lubricants can also affect the deflection temperature of rigid PVC (any lubricant either liquid r having a melting point below HDT of rigid PVC can cause it).
3. Lubricant can have effect on appearance such as on color , gloss and clarity e.g. hydrophilic lubricants can increase water absorption of the PVC compound and a resultant reduction in clarity.
4. Lubricant can also exert a marked effect on heat stability. Generally, they will exert appositive influence by lower in the stock temperature e.g., metallic soaps can function as stabilizers, glycerol, monoesters acts as chelators, while others can interact with the stabilizers.

5. Lubricants, since they have low compatibility and can migrate to the surface, can affect the surface characteristics of the PVC compound. On the positive side, some lubricant will impart slip and anti blocking characteristics to flexible PVC. On the other hand, excess exudation of the lubricant will cause problems in lamination heat sealing, embossing and printing. The lubricant can also influence the compatibility and exudation of secondary plasticizers e.g., in low cost electrical insulation compounds containing CP secondary plasticizer, normal and dibasic lead stearate will permit higher levels of CP's to be used without exudation than the levels allowed by the less compatible paraffin or polyethylene wax lubricants.
6. The lubricants have a marked effect on electrical properties of polymers also. Generally, the more polar or ionic the lubricant, the poorer are the electrical properties. Further differences become greater with an increase in temperature. As 70°C normal lead stearate is two fold better than stearic acid, while a paraffin wax, being less polar, is two fold better than lead stearate. In addition to VR and the related insulation resistance of wires, surface resistivity can be affected by lubricants. The more polar lubricant will reduce surface resistivity and provide some anti static benefits.

2.5 VARIOUS OTHER MODIFYING ADDITIVES:

FLAME RETARDANTS: Although PVC is inherently flame retardant due to its high chlorine contents (approximately 57%) in unplasticized form but when PVC is compounded with diester plasticizers and modifiers, the resultant compositions will be combustible. Flam retardency is restored by adding various solid or liquid additives, chiefly those containing chlorine, phosphorus and antimony an ideal flame retardant should be easy to incorporate in and be compatible with the plastic (i.e., not bleed out), and not alter its mechanical properties. Further more, it should be color less, exhibits good light stability, and be resistant to aging and hydrolysis. It should also be matched to the decomposition temperature of the polymer, i.e., its effect must begin below the decomposition temperature of the plastic and continue over the whole range of its decomposition. It must not cause corrosion, must be temperature resistant, effective in small amounts, odourless and without harmful psychological effects. It must also emit very low levels of smoke and toxic gases and finally be as cheap as possible. Antimony oxide is the widely used flame retardant with usual range of 2-3 phr. Some other examples of commonly used flame retardants are:-

- Chlorinated hydrocarbons, e.g., chlorinated paraffins with 40-70% chlorine.
- Zinc borate and modified barium meta borate.
- Transparent flame retardants (organic phosphates, e.g., tricresyl phosphate).
- Tri phenyl stibene or tri phenyl antimony.

SMOKE SUPPRESSANTS: - The severe problem produced by smoke includes inhalation of toxic particulates, disorientation and panic, eye irritation and obstruction of vision. In case of plasticized PVC, many if not all, of the commonly used flame retardant additives, although effective in controlling flaming combustion, actually increase the amount of smoke produced when the material is incinerated in fire. Even unplasticized PVC, considered to be inherently flame retardant, has been found to produce significant quantities of smoke even during the processing.

Some examples of smoke suppressants are mixtures of iron powder and molybdenum oxide or iron powder, molybdenum oxide and copper oxide; alkali metal zinc ferrocyanides, potassium zinc cuprocyanide, vanadium oxides etc.

ANTIOXIDANTS: Antioxidants prevents the oxidative degradation of polymers. Antioxidants can be hydroquinone, hindered phenols, phosphites, thioesters, epoxies, or aromatic amines. The primary antioxidants used in wire and cable are hydroquinones and hindered phenols. The hydroquinones are more effective as antioxidants but can interfere with peroxide cures. They should be used only in sulphur cured systems. Hindered phenols are not as active as hydroquinones and can therefore be used in peroxide systems. Phenols also give better electrical properties. Volume resistivity is 10 times better with hindered phenols than that with hydroquinones.

COLORANTS: Vinyles may be colored internally through the incorporation of pigments or dyes, or externally through the application of coatings. dyes are organic substances which are soluble in the compound. These materials find limited use in vinyl as well as in most other thermoplastic except at very low concentrations. Major disadvantages are their tendency to migrate and

ease of extraction which is particularly serious with plasticized PVC. Heat resistance is also generally poor.

Pigments may be divided into two major categories as follows:

Inorganic: - this group includes TiO_2, CrO_2, ultramarine blue, molybate orange, metallic powders and flakes. These pigments are characterized by excellent heat resistance low cost in comparison to organics and high opacity.

Organics: - These includes phthalocyanines, quinacridones and sodium benzidines. They have excellent brightness and brilliance

A variety of considerations, such as color, processing temperature, processing ingredients service and exposure requirements transparency, and cost generally narrows the selection of colorants, which are suitable for an application.

3.INSECTS ATTACKING ON PVC:

Deterioration of PVC by microorganisms, fungi, and bacteria is probably the most commonly recognized form, but attack by living organism of higher order, such as insects, rodents, birds, mollusks, and crustaceans is not known.

Deterioration by fungi and bacteria is chemical, involving changes in composition and breaking chemical bonds. It appears to be caused by the action of enzymes, which the micro organism reduces, on additives in the formulation, especially to the plasticizers. Of the two, fungal deterioration seems to be the most prevalent and has received the most attention. This may be because fungi frequently cause discoloration, while bacterial damage is less readily. Other manifestations of microbial degradation include changes in odour or weight, embrittlement, exudation, loss of tensile strength, loss of elongation, and changes in electrical properties. Additive chemicals used to prevent degradation by micro organisms are collectively called microbicides, and include fungicides to kill fungi, and bactericides which kill bacteria (the term microbicide is often used in industry to include chemicals that inhabit growth but don't kill. The proper terms for these kinds of agents are fungistat and bacteristat.). The usual concentration of microbicides ranges from 0.1% to 5%, based on weight of plasticizer.

End products known to have been damaged by microbes include upholstery, wall covering, floor covering, wire and cable, tents, baby pants, outerwear and shower curtains.

Damaged by macro organisms is cheaply physical rather than chemical it involves gross destruction, such as gnawing or boring of holes, instead of molecular changes and the rupturing of chemical bonds. Birds

insects and rodents can penetrate when it represents a barrier to a source of food

Although PVC itself is not susceptible to microbial attack as far as is known, some of the modifying agents used with it provide a nutrient source for both bacteria and fungi. The modifiers known to be microbial nutrients include some lubricants and plasticizers in particular.

It has been reported that rigid and semi rigid PVC are immune to penetration by termites, but attack has occurred on plasticized compounds. The degree of termite penetration varied with plasticizer concentration and structure. Susceptibility increased with increasing level of plasticizer, and decreased when tricresyl phosphate was used. Other effective means of control are the incorporation of the insecticides aldrin and dieldrin, and incorporation of certain inorganic fillers.

PVC compound such as cable jackets and food packages have been attacked by rodents, specially by rats and mice. Damage to cable is apt to occur if they are placed in locations that block the passage of rodents to food sources.

All the insecticides should be chosen on the basis of a suitable combination of efficiency, use-cost, lack of danger to humans, and lack of side effects on the materials being protected.

4.PROCESSING AND PREPARING OF TEST SAMPLES

4.1 **RAW MATERIALS:** -After going through number of PVC formulations from the chemical abstracts and studying the role of various additives, from number of literatures, incorporated in PVC during compounding, the following formulation was developed for purpose of making laminates for testing. Antitermite compounds were selected after thorough literature review and market survey.

RAW MATERIALS (In Grams) USED IN DIFFERENT BATCHES:

Table

Batch no. →	P00	P01	P02	P03	P04	P05	P06	P07	P08
Raw materials ↓									
PVC resin	300	300	300	300	300	300	300	300	300
Tri basic Lead Sulphate	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6
Di Basic Lead Sulphate	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4
Stearic Acid	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
Titanium Oxide	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6

Calcium Stearate	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8
Calcium Carbonate	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
C- Black	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Antifungal Agent	3.375	3.375	3.375	3.375	3.375	3.375	3.375	3.375	3.375
Antirodent Agent	1.125	1.125	1.125	1.125	1.125	1.125	1.125	1.125	1.125
Diisooctyl Phalate	120	120	120	120	120	120	120	120	120
Chlor paraffin Wax (CPW)	60	60	60	60	60	60	60	60	60
Anti termite compound	0.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0

Table

Batch nos. → / Raw materials ↓	P09	P10	P11	P12	P13	P14	P15	P16
PVC resins	300	300	300	300	300	300	300	300
Tri basic Lead Sulphate	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6
Di Basic Lead Sulphate	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4
Stearic Acid	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
Titanium Oxide	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
Calcium Stearate	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8
Calcium Carbonate	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
C- Black	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Antifungal Agent	3.375	3.375	3.375	3.375	3.375	3.375	3.375	3.375
Antirodent Agent	1.125	1.125	1.125	1.125	1.125	1.125	1.125	1.125
Diisooctyl Phalate(DOP)	120	120	120	120	120	120	120	120
Chlorparaffin Wax(CPW)	60	60	60	60	60	60	60	60
Anti termite compound	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0

ANTI TERMITE COMPOUNDS USED FOR VARIOUS BATCHES
Table

Batch nos.	**Anti termite compound**
P01	Creosote oil
P02	Trichlorobenzene
P03	Chlorpyriphos
P04	Lindane
P05	Pentachlorophenol
P06	B P – Wood Protector
P07	CNSL (Cashew Nut Shell Liquid)
P08	Pentaphenepale
P09	Creosote oil
P10	Trichlorobenzene
P11	Chlorpyriphos
P12	Lindane
P13	Pentachlorophenol
P14	B P – Wood Protector
P15	CNSL (Cashew Nut Shell Liquid)
P16	Pentaphenepale

4.2 COMPOUNDING (DRY BLENDING) ON RIBBON BLENDER

Dry blends are mixtures of vinyl resins and other compounding ingredients prepared under sufficient shear and heat to allow the resin particles to take up the other compounding ingredients and form a free- flowing powdery blends. They may be produced by ribbon blenders, high- intensity dry mixers, or other variations of powder mixing equipment. Steam- jacketed ribbon blender was used for making all the formulations.

Ribbon blender is a light duty blender mainly used for easy to mix powder components which are processed like dried granules, pre-sieved powders etc. it is a low shear mixer and mostly used for solid/ solid mixing. Solid/ liquid mixing can also be achieved when high shearing force is not desired. It occupies less head room space for large volume unlike Double cone, 'V' blenders etc. Ribbon blender used was of 'U' shape for low to medium volume capacity with batch size based on 70% of blender volume. Feeding was directly from the top of the blender with capacity available as 500L with matching motor sizes upto 75hp(motor horse power needs will have to be determined for the product to be mixed).

The mixing procedure is not usually critical, but several basic rules should be observed. In ribbon blenders, the resin and other dry ingredients, with the exception of solid lubricants, are added first and allowed for 7 to 10 minutes in order to break up agglomerates. Heat should be used only high plasticizers levels, or in the case of polymeric plasticizers, to make the resin more absorptive. After incorporation of plasticizers and other solid lubricants and other liquid

compounds it is run for 10 to 15 minutes more until it becomes free-flowing powder. Afterwards, the powder is taken out. As such no firm rules are established, since conditions vary among resin properties and mixing intensities of blenders, but in general, a good dry blend should be produced in 20 – 30 minutes.

Among the limitations of ribbon blender are limited use of blender capacity to about half, lengthy mixing cycles, relatively poor plasticizer absorption with highly plasticized formulations or when addition of polymeric plasticizers are required, and comparatively poor dispersion of small amounts of liquids in rigid blends (such as tin stabilizers).

4.3 TWO- ROLL MILLING (MAKING OF SHEETS)

Two- roll mill is a high intensity, high- shear batch- type mixer and has been the basic processing equipment in rubber and plastic processing plants for decades. Two roll- mill is employed exclusively in the compounding of thermoplastics. These are used for additive compounding plastication and homogenization. The resultant product is a sheet that can be either granulated after cooling or fed in plasticated form directly to processing equipment. The simple and versatile design of roll mills makes them very convenient, especially for experimental compounding. They consist of two rolls that generally rotate at different speeds. It provides a very high- localized shear by drawing the mixture into the nip of closely spaced parallel rolls. The viscosity of the matrix can be held high to aid dispersion

because of the extensive surface area for heat transfer. The material passing through the nip is returned to the feed point by rotation of the rolls. A temperature difference between the rolls frequently causes the material to selectively adhere to one of the rolls. The speed of rolls of roll mill used was 27 rpm .

The steps taken for processing on roll mill were as follow:

- Before starting the process, first clean the rolls with a cotton cloth wetted in acetone.
- Turn on the mill and put a normal thermometer in between the rolls to measure the temperature.
- On 80 ^{0}C wash the rolls' surfaces with stearic acid which also acts as external lubricant.
- On 120 ^{0}C (the temperature required for a flexible system) first flush out the rolls with uncompounded PVC resin in small quantity to remove off any foreign particles or impurities. While doing this set the nip for required thickness.
- Now start pouring the compounded PVC powder in the gap between the rolls at their centers. Initially, the entire material does not stick to the rolls and may fall down below over the tray. Pour that material again in nip. Gradually, the material starts sticking to the roll and starts to form a sheet. Keep on cutting that sheet across the roll and put it back again in the gap for homogeneous dispersion.
- Repeat the process till a well- formed sheet is prepared. It generally takes 8- 10 minutes.

- Now, remove off the sheet from the roll without letting it gets folded.

In case during the process if there is some drop in temperature, the material starts falling; so again switch on the heaters to bring the temperature back to 120oC. Now, these sheets are prepared for making the laminates on compression molding machine.

4.4 COMPRESSION MOLDING (MAKING OF LAMINATES):

Compression moulding is a process generally employed thermosets, although for special applications where the technique has particular advantages, it is also used for thermoplastics. The advantage of this process is that the waste and finishing costs are low. The PVC resins used must flow readially when subjected to heat and pressure. Vinyl chloride- vinyl acetate copolymers, such those containing approximately 15%vinyl acetate, are most suitable. Compression moulding may also be used for large sections, which are difficult to form, by other techniques. Floor tiles may be produced by this method using dry blend or plastisols. The production of rigid vinyl sheet for use in automobile windows has also been reported up to 1 inch with excellent clarity and impact resistance may be produced.

The main work on compression moulding machine (with horse power of 5 hp, tank capacity of 150 liters and maximum pressure applied of 150 kg/cm^2) was to make PVC–jute-PVC laminates of all the batches. The following steps were taken:

1. Set the temperature of both the platens as 150°C, which takes 15 to 20 minutes to come on automatic Nuchem comperssion moulding machine.

2. Put the glazed sheets in between the platens, having two pieces of jute (10x10cm) with PVC sheets on both the sides (of the jute pieces).
3. Now up the ram to compress the glazed sheets.
4. When both the platens just compress the sheets with out any additional pressure applied; keep it as such for five minutes.
5. After five minutes increase the pressure to $7N/m^2$ ($35\ kg/cm^2$) (according to the calibration of the machine) and keep it for further one minute.
6. After one minute release the pressure, down the ram and remove the glazed sheets off the machine.
7. After cooling the glazed sheets take the prepared laminates out.

For anti rodent testing the PVC-jute-PVC laminates of size 10x10 cm^2 were prepared. For anti termite testing the PVC-jute laminates of size 1foot x1foot were prepared with the same technique.

Special Observations:

- When the temperature around 160 to 180°C—yellowing of the sheet occurred due to degradation.
- When the temperature was around 130 to 135°C—flow marks developed due to improper fusion and flow of material.

The ideal sheet was obtained at 145 to 150°C.

5. EXPERIMENTAL DETAILS:

5.1 ANTI TERMITE TESTING:

For anti termite testing prepare the one sided PVC jute laminates of 1 foot x 1foot. Then they were rolled with PVC side outwards and then stapled according to testing, the samples were to burry in the ground (with some space in between), up to half of there length around a tree stem which has the termite colonies and proper fencing was done so that animals don't harm the samples. This was kept under observation for 21 days. After 21 days the samples were removed and observed keenly for any attack by termites. If termites have not attacked on them means the samples are passed other wise failed.

This testing was carried out in two phases. In the first phase seven samples with a control sample (which does not have any anti termite compound) were placed on 10th of June and removed on 31st June. In second phase nine samples with one control sample were placed on 12th of July and removed on 2nd of august. The phase testing was carried out around a tree in Shriram institute compound itself and the second phase testing was carried out on the *pahari* situated in front of the Shriram institute.

5.2 ANTI RODENT TESTING:

This testing was carried out on wild rats. The rats were put under starvation for 24 hrs and the samples, i.e., packets of PVC-jute-PVC laminates containing biscuits, were placed for 18 hrs in the rat cage on every usual testing and were observed afterwards. Generally, the samples were placed on 5 pm and removed on 11 am next day. If the rats damage the sample and eat the biscuit inside, the test fails otherwise it passes.

5.3 THERMAL STABILITY TEST (IS: 10810(Part 60)-1988)

The thermal stability is an accelerated method to determine the rate of degradation of PVC compound with time under the influence of temperature. Compounds with high thermal stability can with stand continuous processing temperature and it is an indication of performance at normal and elevated temperature in actual usage.

Thermal stability is the indication time in minutes for the PVC compound at 200^0C, after it has been kept in the test apparatus. This is indicated by the red coloration on pH paper produced by the evolution of hydrochloric acid.

Apparatus:

(i) Thermostatically controlled heating apparatus at 200± 0.5 ^{0}C .

(ii) Glass test tubes- 110 mm long with an outer diameter of approximately 5mm and inner diameter of 4.0mm.

(iii) Stop watch

(iv) Thermometer with least count of 0.1^0C.

(v) Universal indicating paper strips approximately 15mm long and 3mm wide of pH range 1-10.

Test Specimen:

(1) Strips each of mass 50 mg, cut from insulation of sheath to be tested were taken.

(2) No. of specimens taken- three

(3) Material was conditioned at 27 ± 2^0C and relative humidity of 50±5% for 20 hrs.

Procedure:

(1) Each specimen was introduced into the test tube so that it occupied the bottom of the test tube and project not more than 30mm above the bottom.

(2) A strip of dry universal indicating paper was inserted into the open end of the glass tube so that the strip protruded about 5mm out of the tube and is held in position.

(3) The glass tubes were placed into the heating apparatus (for a depth of 60mm), which already attained the test temperature specified.

(4) The time taken for the universal indicating paper to change its color into red was noted down. This time in minutes is the thermal stability of the sample.

5.4 Water Absorption test (ASTM-D570)

This test method covers the determination of the relative rate of absorption of water by plastics well immersed. The test method is intended to apply to the testing of all types of plastics including cast, hot- moulded and cold- moulded resinous products, and both homogenous and laminated plastics in rod and tube form and in sheets 0.13mm or greater thickness.

Apparatus used:

(1) Balance- an analytical balance capable of reading 0.0001gm.

(2) Oven- capable of maintaining uniform temperature of 50 ± 3^0C.

Test specimen:

The test specimen was in the form of a bar 76.2mm long by 25.4mm wide by the thickness of the material having smooth edges free

from cracks. The cut edges shall be made smooth by finishing with fine sand paper. Along with that it was properly conditioned. Overall three test specimen were taken.

Procedure:

The conditioned specimens were placed in a container of distilled water maintained at a temperature of 23 ± 1^0C and rest on edge and be entirely immersed. At the end of 24hrsthe specimens were removed from the water one at a time, all surface water was wiped off with a dry cloth and weighed to the nearest 0.001gm immediately.

Formula used:

Increase in weight, % ⟶ Wet weight – Conditioned weight

Conditioned weight

5.5 Tensile strength test (ASTM D 638M- 93):

Tensile strength in a broad sense is measurement of the ability of a material to with stand forces that tend to pull it apart and to determine to what extent the material stretches before breaking many plastics are very sensitive to the rate of straining and environmental conditions. Therefore the data obtained by this method can not be considered valid for applications involving load- time scales or environments vary different from this method.

Scope:

(1) This test method covers the determination of the tensile properties of unreinforced and reinforced plastics in the form standard dumbbell shaped test specimens when tested under defined

conditions of pretreatment, temperature, humidity and test machine speed,

(2) This test can be used for testing materials of any thick ness upto 14mm.material with thickness greater then 14mm must be reduced by machining.

Apparatus:

The tensile testing machine of constant rate of cross head movement is used. it has a fixed or essentially stationary member carrying one grip and a movable member the second grip. A controlled velocity drive mechanism is used. A load indicating mechanism capable of indicating total tensile load with an accuracy of 1% or better is used. An extensometer is used to determine the distance between two designated points located with in the gauge length of the test specimen as the specimen is stretched.

Test specimen and conditioning:

Test specimens can be either injection molded or compression molded. The specimen can also be prepared by machining operations from materials in sheet, plate, slab, or similar form. In our case the specimen is compression molded having a thickness of 3mm.for the specimens of thickness under 4mm type IV type of specimen dimensioned are specified by the standard. The required specimen is cut out with the help of a die.

SPECIFICATIONS FOR TYPE IV SPECIMEN

Table

	Dimensions	Tolerances
	mm (in.)	mm (in.)
W-Width of narrow section	6(0.25)	±0.5(±0.02)
L-Length of narrow section	33(1.30)	±0.5(±0.02)
WO-Width overall	19(0.75)	+6.4(+0.25)
LO- Length overall	115(4.5)	No max.(no max.)
G-Gauge length	25(1.0)	±0.13(±0.005)
D-Distance between grips	65(2.5)	±5(±0.2)

The specimens shall be conditioned using standard conditioning procedures (ASTM D618) since the tensile properties of some plastics change rapidly with small change in temperature, it is recommended that the test be conducted in the standard laboratory atmosphere of 23±2^{0}C and 50±5% relative humidity.

Test procedure:

The speed of testing is the relative of motion of the grips or fixtures during the teat. There are basically different testing speed specified in the ASTM D638 standard. The most frequently employed speed of testing is 0.2 in./ min., whenever possible, the speed indicated by the material being tested should be used. If a test speed is not given, appropriate speed that causes rupture between 30 secs. To 5 min. should be chosen. The test

specimen is positioned vertically in the grips ot the testing machine. The grips are tightened evenly and firmly to prevent any slippage. The speed of testing is set at the proper rate and the machine is started. As the specimen elongates, the resistance of the specimen increases and is detected by a load cell. This load value is recorded by the instrument. Some machines also record the maximum load obtained by the specimen, which can be record after the completion of the test. The elongation of the specimen is continued until a rupture of the specimen is observed. Load value at the break is also recorded. The tensile strength is calculated as

Tensile strength (psi) = Force (load)(lb)/ Cross section area (sq. in.)

The percent elongation is calculated as:

$$\% \text{ Elongation} \longrightarrow$$

$$= \frac{\text{Final gauge length (mm)- initial gauge length (mm)}}{\text{Initial gauge length (mm)}} \text{X100}$$

(5.6) Izod- Impact Strength Test (Cantilever Beam Test)(ASTM D 256 – (93a): -

This test method covers the determination of the resistance of plastics to breakage by flexural shock as indicated by the energy extracted from 'standardized' pendulum- type hammers, mounted in "standardized" machines in breaking standard specimen with one pendulum swing. The notch in the Izod specimen serves to concentrate the stress, minimise plastic deformation, and direct the fracture to the part of the specimen behind the notch. Scatter in energy- to - break is thus reduced.

Any of the failure should be recorded according to following coding.

C – *complete break* – a break in which the specimen separates into two or more pieces.

H – *hinge break* – an incomplete break such that one part of the specimen cannot support itself above the horizontal when the other part is held vertically.

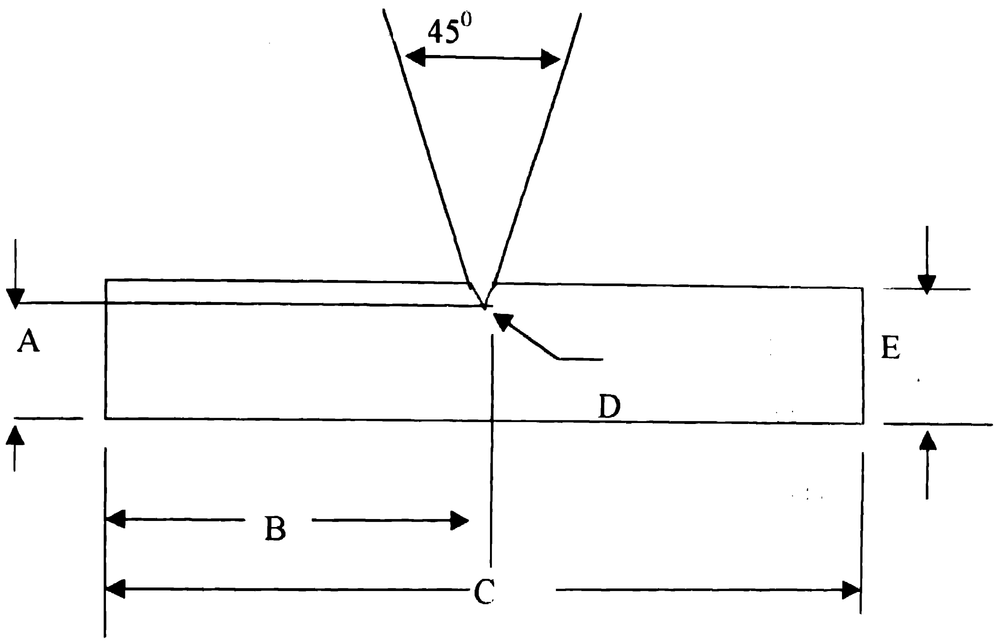
P – *partial break* – an incomplete break that does not meet the definition for a hinge break but has fractured at least 90% of the distance between the vertex of the notch and the opposite side.

NB – *non-break* – an incomplete break where the fracture extends less than 90% of the distance between the vertex of the notch and the opposite side.

Apparatus; -

The machine shall consist of a massive base on which is mounted a vise for holding the specimen and to which is connected, through a rigid frame and anti- friction bearings, one of a number of pendulum type hammers. A pointer and dial mechanism should be there for indicating the excess energy remaining in the pendulum after breaking the specimen. A jig for positioning the specimen in the vise and graphs or tables to aid in the calculation of the correction for friction and windage also should be included. The pendulum shall consist of a single or multimembered arm with a bearing on one end and a head, containing the striking nose, on the other. The effective length of a pendulum shall be between 0.325 and 0.406m so that required elevation of the striking nose may be obtained by raising the pendulum to an angle between 60^0 and 30^0 above the horizontal.

Test Specimens: - The specimen should conform to the dimensions and geometry of the under drawn fig. Except the width of the specimen shall be in accordance with section. While testing it was kept as 3mm.



A = 10.16 ± 0.05 mm

B = 32.00 mm max

31.50mm min.

C = 63.50 mm max.

60.30 mm min.

D = 0.25R ± 0.05 mm

E = 12.70 ±0.15 mm.

The energy to break the sample completely shall be noted, on which basis the impact strength is calculated.

6.RESULTS AND DISCUSSION:

(1) On the basis of antitermite testing (Article no.5.1): -

Out of all samples i.e. from P00 to P16 some samples got pale yellow stains in the form of patches, some were attacked by termites and all have got faded in their respective white colors due to Sunlight and moisture of ground. The description can be given as underwritten.

P00, P01, P03, P09 and P12 have got the patches of pale yellow color and were severely attacked by termites.

P04, P08, P12 and P16 got the large yellow-patches on two or three places on respective samples with an attack of termite up to some consideration.

P06, P07, P14 and P15 got the patches of yellow color and were nimbled by termites badly. Along with that these laminates were found with looseness between PVC sheet and jute.

P02, P05, P10 and P13 got only very small patches on some places. No termite attack was observed.

The yellow color patches were the result of fungal attack which was least found in P02, P05, P10 and P13. It means these samples are best resistant against fungal attack.

(2) On the basis of antirodent testing (article no 5.2): -

The tests were carried out according to prescribed method and following table was prepared, in which P indicates for pass and F indicates for fail.

P01	P02	P03	P04	P05	P06	P07	P08	P09	P10	P11	P12	P13	P14	P15	P16
P	P	P	F	P	F	P	P	P	P	F	P	P	F	P	P
P	P	P	F	P	P	F	P	F	P	P	P	P	F	P	F
P	P	P	F	F	P	F	P	P	P	F	F	P	P	F	P
P	P	P	F	P	F	P	F	P	P	P	F	P	P	F	P
F	P	F	F	P	P	F	F	F	P	P	F	P	P	F	F

On keen observation of the table it can be seen that the samples P02, P10 and P13 are all passed and P01, P03 and P05 are failed in one test only. So, out of these six samples the final sample can be chosen.

(3) On the basis of thermal stability test: -

After conducting the above two tests this test was performed only for samples P02, P10, P13, P01, P03 and P05 according to the test procedure described in article 5.3. All the samples passed the test successfully with in the prescribed limits i.e. 40 to 60 minutes.

(4) On the basis of water absorption test: -

This test was also performed only for samples P02, P10, P13, P01, P03 and P05 according to the test procedure described in article no. 5.4. All the samples passed the test within specified limits i.e. they did not absorb water more than 0.4- 0.6 % of their weight.

(5) On the basis of tensile strength test: -

This test was also carried out only for samples P02, P10, P13, P01, P03 and P05 according to the test procedure described in article no. 5.5. All the samples passed the test within specified limits i.e. 40-60 MN/m^2. Percentage elongation was also under limits.

(6) On the basis of Izod- Impact strength test: -

This test was also carried out only for samples P02, P10, P13, P01, P03 and P05 according to the test procedure described in article no. 5.6. All the samples passed the test within specified limits i.e. between 2-10 KJ/m^2.

Conclusion: -

After examining the results of all the samples tested, it can be inferred that only six samples i. e. P02, P03, P01, P05, P10 and P13 are of worth. Out of which only four samples i. e. P02, P05, P10 and P13 passed the antitermite test. As far as antirodent test is concerned there are only three compounds which passed all the five tests against wild rats i.e. P02, P10, and P13. Rest three i.e. P05, P03 and P01 failed one time out of total five times.

Now, if P02, P10 and P13 are considered along with their other properties as thermal stability, water absorption, tensile strength and Izod impact strength; we can see that they are quite compatible with those tests as the formulation was guided by Dr. M.Q Parwez on the basis of experience and I will have to check whether any difference comes on mixing the antitermite and antirodent compounds in the formulation. And obviously there was no change on the physical properties.

Again on consideration of P02, P05, P10 and P13 - P02 and P10 have the same antitermite compound, the only difference is the concentration of the amount. Same is the case with P05 and P13, but P05 failed one antirodent testing so it cannot be relied on with lower concentration. P13 can be considered fine having higher concentration. Between P02 and P10 as both passed all the tests satisfactorily, we will consider P02 only on the basis of economy as it has lower concentration (half times) as compared to P10.

Further in between P02 and P13 as P13 is having a higher concentration so on the basis of economy P02 can again be chosen.

Finally, it may be concluded on the basis of above discussion that P02 is the best suitable compound having antitermite compound as Trichlorobenzene and antirodent compound as Zinc phosphide with specified concentration percentage of PVC resin. On unavailability of P02 in

the market P13 can also be used (taking time as a factor) with antitermite compound as Pentachlorophenol and antirodent compound as Zinc phosphide with specified concentration as described in the article 4.1.

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